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InnerSea Technology

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The goal of the Insulating Biomaterials work is to identify and evaluate materials, coatings, and assembly techniques suitable for protection of integrated circuit devices being considered for neural prosthetic applications.

Instrumentation Systems

Accelerated detection of degradation is the main tool for studying materials for implantable devices. The new Passivation Test System consists of 4 major components: the Tube Top, the Measurement Unit, the Data collection Unit, and the Calibration Unit. These components are described below. Basically, as illustrated in Figure 1 the device to be tested is placed into the saline soak tube.

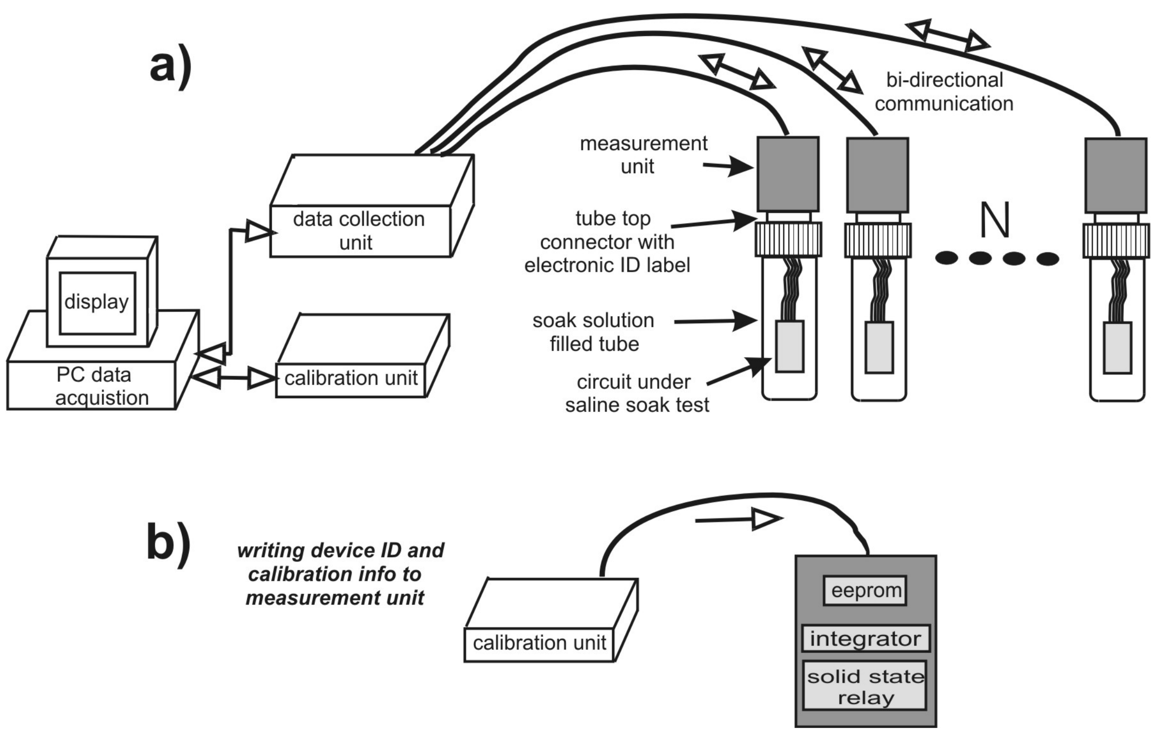


Figure 1: Cartoon showing basic elements of new test system.

1) the Tube Top - This provides: a physical attachment point for the device under test; the electrical connections to the system; and an EEPROM that contains information about the particular device in that is under soak test in that respective tube.

2) the Measurement Unit - This contains: the analog circuitry required to perform the measurement; a calibration check unit; a continuity tester for triple

track devices; and another EEPROM which contains required calibration information.

3) the Data Collection Unit - This accepts the data (consisting of timing signals) from the measurement units, converts them into leakage values, and transmits those values to a host computer.

4) the Calibration Unit - This is a standalone unit that is used with each device before any testing at all is performed, and it is normally used only at the onset of testing. Thus, if a device is tested each day for 100 days, the Calibration Unit is “hooked” up to Measurement Unit at the start of day 1 so that device identification data can be downloaded to the EEPROM that is located on the Tube Top, along with the calibration information particular to the device under test and that will be needed for the Measurement Unit to interpret the measured parameters for every succeeding test day.

Recent Results:

During this period the Passivation Test Integration system was further refined and debugged. The first set of 32 production Measurement Units (MU’s) were fabricated for us by Nexis Custom Electronics of Woburn, Massachusetts. This was our first experience with a contract manufacturer, and we found a short learning curve involved with this. Overall the experience was great, and Nexis did a wonderful job on the assemblies. Unfortunately, we will be unable to utilize them again, as they are in the process of closing their Woburn facility. We found it was advantageous to have the assembly house nearby, to quickly deal with any unforeseen problems. After the assemblies were completed we discovered three small errors in the design of the MU. We were able to rework the units easily to correct these problems.

The sticking point on the system has been the complex Data Collection Unit (DCU). Three major problems have been discovered:

1. In the original design, there was no way to reset and individual MU at the beginning of a test point. The idea was we would just ignore the first trigger

received from a unit, and instead begin taking data when we saw that first trigger. This proved to be unworkable since it would require a complex additional set of logic just to track this first trigger pulse. Instead a much simpler method, where the MU would receive an integrator reset at the beginning of a test point, was implemented. The reset is now addressable for each MU, and a fixed width pulse is generated by the system to perform the integrator reset.

2. In addition to the integrator reset not being addressable, when the reset occurs, it causes a trigger pulse to be sent from the MU to the DCU, causing the DCU to think the measurement has completed. This resulted in corrupt measurement data. A redesign of the internal logic of the FPGA to ignore triggers that occur during a reset cycle corrected this problem, but it did take a considerable amount of time to track down this bug.

3. Once the logic problems were solved a rather nasty problem showed up. Plugging and unplugging the MU's from the DCU may cause a transient that can damage both units. Since the whole point of the system is to have the flexibility to move jars around, we need a robust interface to allow this to happen without problems. This is the issue we are currently working on. A modified Hot Swap interface is being designed to eliminate this problem.

FR4 Printed Circuit Board Technology

While most of the focus of the Insulating Biomaterials contract is on the encapsulation and packaging of long term implantable integrated circuit technology, a number of groups are also using more traditional printed circuit board technology to good advantage with the ready availability of advanced, miniaturized commercial integrated circuits. In addition, and of direct relevance to this program, a support substrate for holding power supply conditioning circuitry, signal devices, and batteries is needed for long term animal testing. However, FR4 technology was not designed to function for long times when immersed in saline solutions. Accordingly, a set of test devices was designed and variations of typical processes were insulated with several materials commonly used for high reliability packaging, and also with silicone.

Six different types of circuit board (FR4IDEA050628) with printed inter digital electrodes (IDE) from Advanced Circuits are examined. The following describes the board construction:

"G" type boards were fabricated with gold/nickel plated copper traces. These boards were coated with solder mask material everywhere except for a square area defining the IDE.

"GM" type boards also were fabricated with gold/nickel plated copper traces and were coated with solder mask material everywhere except for the traces defining the IDE.

"SM" type boards were initially fabricated with copper traces and coated with solder mask material everywhere except for the traces defining the IDE. These traces were then plated with solder.

"NM" type boards were fabricated with solder plated copper traces with no solder mask at all on the boards.

"FM" type boards were fabricated with copper traces and coated with solder mask material everywhere.

"BT" type boards were fabricated with copper traces. The tops of the boards were sealed with a thin layer of FR which is the same material used for circuits boards.

While FM and BT boards were tested "as is", the G, GM, SM, and NM boards were coated with three different types of polyurethane (1122, 1A20, and 1A33) from HumiSeal as well as MED4-4220 silicone from NuSil for comparison. All the boards were then slid through precut slots on test jar's green caps and were fixed with MED2-4220 quick cure silicone at a position such that only coated portion will be in the jar and 20-pin socket connectors were outside. Adaptor boards for 20-pin connector to DB25 connector were finally attached and the devices were ready for pass test measurement. On 9/2/05, the samples were put under dry test. After dry measurement, PB ringer was added to the test jars on 9/7/05. The experiment process and results are summarized as follows.

PCBHUMISEAL-1122-G, PCBHUMISEAL-1122-GM, PCBHUMISEAL-1122-SM, PCBHUMISEAL-1122-NM

G, GM, SM, and NM boards were dip coated with polyurethane1122. Drying overnight before handling, PCBHUMISEAL-1122 samples were then cured at 75^0C for 30 minutes. Dry measurement showed that the G, GM, and SM devices had resistance at $10^{10}\Omega$ and the device with no mask (NM) at $10^9\Omega$ respectively. After soaking, all PCBHUMISEAL-1122 devices failed on first measurement. While corrosion was more confined at IDE section for Au/Ni plated samples, corrosion was extended from IDE to the rest of leads, especially to the leads with bias for SM and NM devices.

PCBHUMISEAL-1A20-G, PCBHUMISEAL-1A20-GM, PCBHUMISEAL-1A20-SM, PCBHUMISEAL-1A20-NM

G, GM, SM, and NM boards were dip coated with polyurethane1A20. Allowing 60 minutes to dry before handling, PCBHUMISEAL-1A20 samples were then cured at room temperature for several more days. Dry measurement showed that the devices of G, GM, and SM had resistance at $10^{14}\Omega$ and NM device had resistance mostly at $10^{12}\Omega$. Although PCBHUMISEAL-1A20 devices were less corroded than PCBHUMISEAL-1122 in general, all PCBHUMISEAL-1A20 devices failed on first wet measurement. Again corrosion was more confined at IDE section for Au/Ni plated samples and extended from IDE to the leads with bias for SM and NM devices.

PCBHUMISEAL-1A33-G, PCBHUMISEAL-1A33-GM, PCBHUMISEAL-1A33-SM, PCBHUMISEAL-1A33-NM

G, GM, SM, and NM boards were dip coated with polyurethane1A33. Allowing 15 minutes to dry before handling, PCBHUMISEAL-1A33 samples were then cured at 75^0C for 30 hours. Dry measurement showed that the devices of G, GM, and SM had resistance at 10^{12}-$10^{14}\Omega$ and NM device mostly at $10^{11}\Omega$. While most of PCBHUMISEAL-1A33 devices failed on first wet measurement and corrosion pattern was very similar to PCBHUMISEAL-1A20, there are two exceptions — PCBHUMISEAL-1A33-G (E10) measured at $8x10^{10} \rightarrow 7x10^9\ \Omega$ (-5, +5V), and

PCBHUMISEAL-1A33-GM (E10) at $1.9x10^{11} \rightarrow 4.6x10^{10}$ Ω (-5, +5V) after one month wet test. The pass test is currently continuing for these two devices.

<u>PCBMED4-4220-G, PCBMED4-4220-GM, PCBMED4-4220-SM, PCBMED4-4220-NM</u>

For comparison to polyurethane sealing, G, GM, SM, and NM boards were flooded with MED4-4220 silicone. Vacuumed to remove trapped air, PCBMED4-4220 samples were then cured at 125^0C for 3 hours. Dry measurement showed that the G, GM, SM samples had resistance at 10^{13}-10^{14}Ω and NM sample mostly at 10^{12}Ω. After one month wet test, Au/Ni plated devices (G & GM) have maintained mostly at 10^{12}Ω (-5, +5V) while SM & NM devices at 10^9Ω (-5, +5V). The pass test is currently continuing for PCBMED4-4220 samples.

<u>PCBFR4IDEA050628-FM, PCBFR4IDEA050628-BT</u>

The last two sets of board, FM & BT, were not coated and tested “as it is”. Dry measurement showed that both types of devices had resistance at $10^{11}/10^{12}$Ω. After soaking, FM device failed on first measurement and corrosion was extended from IDE section to the leads with bias. A further examine under microscope revealed pinholes in solder mask over copper traces. After one month wet test, BT device has maintained mostly at 10^{12}Ω (-5, +5V) and pass test is currently continuing for this device.

In summary, the experiment showed the following observations:

For G & GM boards, corrosion is more confined at IDE section.

For SM, NM, & FM boards, corrosion has extended from IDE onto the leads, especially the ones with bias.

While SM samples are most corroded, NM & FM devices have less degree of corrosion than the rest of types.

Among three types of polyurethane (1122, 1A20, and 1A33) from HumiSeal, 1122 is the worst sealer. The samples coated with 1122 were most corroded in general.

Perhaps surprisingly, MED4220 appears to be performing better than the materials designed for this purpose.

Mechanical Properties of Materials Testing

New Tests Initiated:

Pull tests on silicone tubing & wire using XL110 crosslinker.

Silicone tubing control	544 gms
Silicone tubing + XL110	666 gms
Bay Wire + XL110	89.88 gms
NEEWC + XL110	19.82 gms

Bis(triethoxysilyl)ethylene – pull tests to determine whether using Bis… as a pretreatment on glass slides will increase the adhesion between the slide and MED4-4220 silicone adhesive. Ave. to date = 2922 gms.

FM LCP 517-75 – study using 3 types of pretreatments of LCP from Foster-Miller. The 517-75 samples use A174 with varying pH, hydrolysis and curing times. Early results show a better result with normal pH.

FM LCP 517-76 – Two other samples from Foster-Miller were prepared pretreating the LCP with vinyltriethoxysilane and allyltriethoxysilane. Both show a greater adhesion than the A174 samples, with the vinyl… showing the greater adhesion.

LCP Pretreatment – Bis(triethoxysilyl)ethylene – samples created using Bis… as the pretreatment between LCP and MED4-4220. Ave. pull has been ~ 295 gms.

LCP Pretreatment – N-(2-aminoethyl)-3-aminopropyltriethoxysilane – sample creasted using N-(2-…as a pretreatment between LCP and MED4-4220. Ave. pull has been ~ 875 gms.

MED4-4220 Lot 35070 – Having experienced rapid set times in a previous batch of MED4-4220, samples were created to determine the average pull force of the new lot of MED4-4220. Two samples averages are ~2450 gms and ~2500 gms.

N-(2-aminoethyl)-3-aminopropyltriethoxysilane – to determine whether using N-2… as a pretreatment will increase the adhesion of MED4-4220 to glass slide. Average has been ~ 2789 gms.

XL110 Pretreatment – to determine whether using XL110 as a pretreatment on glass slides will increase the adhesion to MED4-4220. Ave. has been ~ 2936 gms.

Samples that were ended:

MED2000 #2 – Sample created using MED2000 between slide and fiberglass tape to determine its adhesion values. Values varied greatly. Ave. = 2039.98 gms. Std. Dev. = 943.93 gms.

MED1511 #1 – Sample created using MED1511 as the adhesive silicone between quartz slide and fiberglass tape. Ave. = 1885.9 gms. Std. Dev. = 304.02 gms.

Teflon Tape – sample created using Teflon tape bands across slide to separate pulls. The average pull was 1055 gms. with a std. dev. of 266.19 gms.

MED4770 + CSM4220-3 – study to see if a slab of MD4770 would bond with significant force to CSM4220-3. Ave. force ~ 293 gms.

Adhesion Test 020725 – test to determine the adhesion and durability of .007 cm fiberglass tape. Previous samples had bee constructed with .003 fiberglass tape and some samples had evidenced ripping and a lack of integration of silicone. The larger weave was thought to reduce these problems. Ave. force = 790 gms., Std. Dev. = 161.62 gms.

Changes:

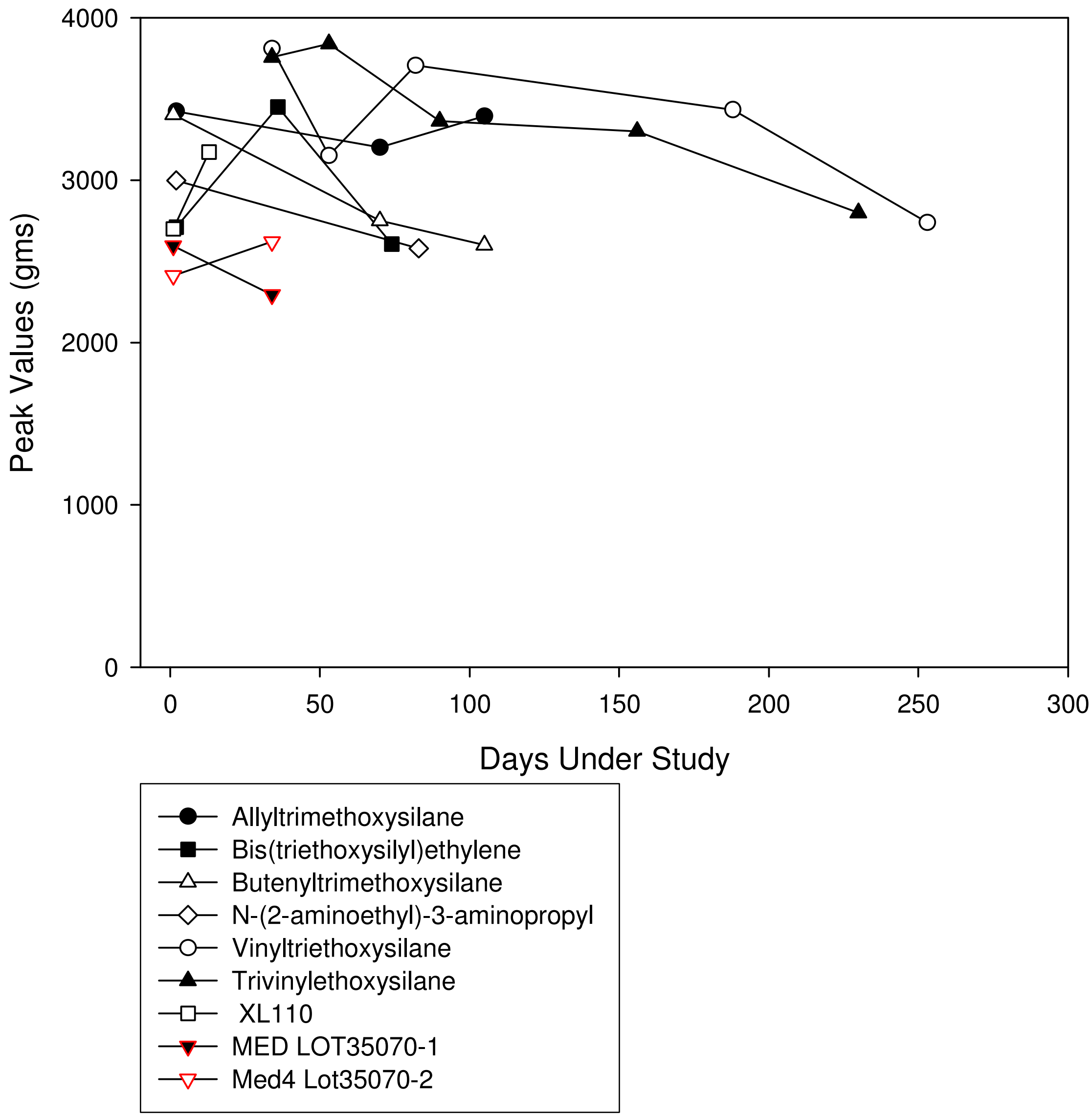
Uptake studies – Several samples have shown a loss of weight in the studies involving Lactic Acid, Albumin and NaCl. Normalized weight change in some of these samples has been between -.002 and -.005 gms.

<u>Interim Results for Silicone/Glass Bonding with Adhesion Promoters</u>

A crucial aspect of use of silicones on silicon dioxide surfaces for long term protection of integrated circuits and systems is the mechanical adhesion. If adhesion fails, then the interface will hydrate and the device will fail. While long term interdigitated finger electrode array testing can detect loss of electrical integrity, prior to that, loss of adhesion will occur. Thus this is a form of accelerated testing that does not rely on fantasies of reliable projections. Rather, it directly measures the failure mechanism.

Below is a summary graph of adhesion promoters in current use and early results. While trends are apparent, it is still too early to determine if those are real or simply process variations.

Silane Pretreatments
Glass slides, large weave fiberglass tape, MED4-4220 LC



CVD Deposition of Silicones

Since silicones seem to perform indefinitely as protective coatings for electronics submerged in saline environments under some conditions, and microstructures are the primary target for the encapsulation technology and techniques, substantial effort has been directed towards development of a gas phase, thin film deposition process. Recent work is summarized below:

Work during July and August focused on the deposition of wire coating that would be both flexible and electrically resistive. This had been previously unachievable due to the inability to sufficiently cool the freestanding wire substrate within the reactor. To avoid this issue, wire coatings were constructed by placing wires in direct contact with the cooled substrate stage. Complete coating of the wires was then achieved through a two step process doing first one side of the wire then the other. Azo-t-butane was utilized as the radical initiator along with the normal Trivinyl-Trimethyl-Cylcotrisiloxane monomer. Deposition rates on the order of 50-75nm/min were observed on a reference silicon wafer placed in the reactor while the wires were coated. Total coating thickness (again measured by reference wafer) was between one and five microns. Coatings were observed to be electrically resistive when wire samples were placed in a saline bath and an electrical bias applied across them. Coatings maintained resistivity for up to 24 hours of soak time. Commencement of long term soak testing of coated wire samples is planned in the near term.

In order to optimize the electrical properties of the material, a new electrical measurement apparatus was constructed for the characterization of thin film resistivity. The apparatus consists of a double shielded metal box along with a highly sensitive Keithley 617 electrometer. When appropriately grounded, the electrometer can measure resistance of up to 1012 Ohms, which translates to resistivity of up to 1015 Ohm-cm. Measurements from this apparatus confirmed the Innersea data that the deposited coatings possessed resistivity on the order of 5 X 1015 Ohm-cm. In addition, variations in bulk resistivity of up to two orders of magnitude have been observed with variation of deposition speed, initiator, and temperature. The source of these variations will be investigated in the near term. In addition, wire samples will be prepared utilizing conditions optimized for coating resistivity and their properties will be characterized.

A poster detailing this work was prepared and presented at the 2005 NIH Neuroprosthetics workshop in September.